ANALYST		LAB CODE	

Parameter: Base/Neutrals and Acids (GC/MS)
Checklists
04/02

INSTRUMENT _____ DATE _____

METHOD OF ANALYSIS

	EPA 625 Series, 40 CFR, Part 136
	18th Edition of Standard Methods 6410

	APPARATUS AND MATERIALS	**Y**	**N**
1.	Is glassware heated at 400ºC (15-30 min.) or rinsed with pesticide grade acetone & hexane? [3.1] **NOTE**: Volumetric glassware should not be heated.		
2.	Are KD concentrator tubes calibrated (calibrations checked) at the volume/s employed? [5.2.3]		
3.	Have boiling chips been heated to 400ºC for 30 min. for Soxhlet extract with methylene chloride? [5.3]		
4.	Is splitless injection employed for capillary columns? [5.6.1]		
5.	Is the data system capable of extracted ion current profile (EICP) plots--a plot of m/z vs. time? [5.6.6]		
	SAMPLING AND STORAGE		
6.	Are samples collected in glass containers (amber preferred) with lids lined with Teflon or foil? Automatic sampling equipment must be as free as possible of Tygon tubing. [9.1]		
7.	Are samples kept at 4°C from time of collection until extraction? [9.2]		
8.	Were samples dechlorinated using sodium thiosulfate at time of collection? [9.2]		
	EXTRACTION - Separatory Funnel		
9.	Is extraction completed within 7 days of collection and completely analyzed within 40 days of extraction? [9.3]		
10.	Is a reagent water blank processed with each set of samples or when reagents are changed? [8.1.3]		
11.	Is entire sample extracted? [10.2]		
12.	Is a surrogate standard spiking solution containing at least three surrogates at a concentration of 100μg/mL added to sep funnel prior to extraction with the pH adjusted to >11 using 10 N sodium hydroxide solution? [10.2]		
13.	What surrogates are being used?		
14.	Are samples extracted at pH >11 (NaOH) via three separate extractions--60 ml CH_2Cl_2 each or extracted for 24 hours using continuous extraction? [10.2]		
15.	Are samples extracted at pH <2 (H_2SO_4) via three separate extractions--60 ml CH_2Cl_2 each or extracted for 24 hours using continuous extraction? [10.5]		
16.	Is each fraction dried by passing it through $NaSO_4$? [10.7]		

		Y	N
17.	Are the B/N and A extracts concentrated to about 0.5 ml using Kuderna Danish Apparatus (macro- then micro- Snyder columns)? [10.9]		
18.	Are the Snyder column balls wetted? [10.8]		
19.	Is the hot water bath temperature (60-65ºC) and vertical position of the KDs adjusted to allow completion of concentration within 15-20 min. for 3-ball Snyder and 5-10 min. for 2-Ball Snyder column? [10.8 and 10.9]		
20.	Is final volume of extracts adjusted to 1.0 mL? [10.9]		
	EXTRACTION - Continuous		
21.	Is extraction completed within 7 days of collection and completely analyzed within 40 days of extraction? [9.3]		
22.	Is a reagent water blank processed with each set of samples or when reagents are changed? [8.1.3]		
23.	Is entire sample extracted? [10.2]		
24.	Is pH adjusted to >11 with 10 N sodium hydroxide solution and then transferred to continuous extractor? [11.2]		
25.	Is l mL of surrogate standard spiking solution added to extractor? [11.2]		
26.	Is sample bottle rinsed twice with 50-100 mL of methylene chloride? [11.2-3]		
27.	Is additional (200-500 mL) methylene chloride added to distilling flask along with sufficient water for proper operation and extracted for 24 hrs.? [11.4]		
28.	Is extract properly dried, concentrated and sealed? [11.4]		
29.	Is a clean distilling flask charged with 500 mL of methylene chloride, pH of aqueous phase adjusted to <2 (H_2SO_4), and extracted for 24 hrs.? [11.5]		
30.	Is extract properly dried, concentrated and sealed? [11.5]		
	CALIBRATION		
31.	Are stock standard solutions stored sealed in Teflon-sealed screw-cap bottles at 4ºC (-10ºC suggested)? [6.7.2]		
32.	Are stock standards replaced after 6 months? [6.7.3]		
33.	Does calibration involve a minimum of three conc. with one standard at the minimum reporting limit? [7.2.1]		
34.	Are internal standards used, does it contain at least 3 analytes from each group? [7.2]		
35.	Is the calibration curve verified each working day by analyzing at least one standard (near the expected sample conc.) with recovery of ± 20%? [7.3]		
36.	Before analysis of samples, is a reagent water blank analyzed to demonstrate that system is under control? [8.1.3]		
37.	Are check standards analyzed at a rate of 5% of samples? [8.4]		
	QA/QC		
38.	Have the following operations been performed by each analyst to demonstrate accuracy and precision? [8.2]		
	a.) Were four quality control (QC) check samples containing each parameter of interest at a concentration of 100 μg/L in reagent water prepared and analyzed? These samples must have been obtained from a source separate from the calibration standards used. [8.2.2]		

		Y	N
	b.) Did the standard deviation (s) and average recovery (X) in μg/L meet acceptance criteria listed in Table 6? [8.2.5]		
	c.) If there was a failure, were the four aliquots prepared again and analyzed for the failed parameter? [8.2.6.2]		
	d.) If any parameter failed more than once, were all parameters reanalyzed? [8.2.6.2]		
39.	Are 5% of samples from <u>each sampling site</u> spiked? If only 1-20 samples are analyzed per month, only one spike is required. [8.3]		
40.	What is the concentration of the sample spike? (1 to 5 times background is recommended.) [8.3.1]		
41.	Do spike recoveries meet the acceptance criteria? [8.3.3]		
42.	For each failed spike, was a QC check standard analyzed and acceptable recovery (listed in Table 6) achieved? [8.4]		
43.	Is each sample, standard, and blank spiked with surrogate standard spiking solution (minimum of three surrogate compounds)? [8.6]		
44.	Are the DFTPP criteria in Table 9 met before any sample analysis is performed? [12.3]		
45.	Are the GC columns (packed) performance verified each day? B/N via Benzidine (tailing factor for 100 ng <3.0) [12.4] Acid via Pentachlorophenol (tailing factor for 50 ng <5.0) [12.5]		
46.	Is internal standard added to sample extract immediately before it is injected into the instrument? [13.3]		
47.	Is solvent flush technique used for manual injections, which are limited to 2-5 μL? (Smaller volumes (1.0 μL) may be injected using an autosampler) [13.4]		
48.	Does the retention time of the sample target compound agree within <u>+</u> 30 seconds of that measured for the standards? [14.1.2]		
49.	Do the relative intensities of the three characteristic ions agree within <u>+</u> 20% of the relative intensities listed in Table 4 & 5? [14.1.3]		
50.	Are the compound concentrations in the sample calculated using the response factor (RF)? [15.1] NOTE: If the RF is <35% RSD, the average RF may be used. [7.2.1]		
51.	Are samples diluted and re-analyzed if the measured conc. is above the highest calibration std. conc.? [13.5]		

PROBLEMS: